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The effect of silicon substrate on thickness of SiO_2 thin film analysed by spectral reflectometry and interferometry

P. Hlubina, J. Luňáček, D. Ciprian

Department of Physics, Technical University Ostrava, 17. listopadu 15, 708 33 Ostrava-Poruba, Czech Republic

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Abstract Techniques of spectral reflectometry and interferometry are used for measuring small changes in thickness of SiO_2 thin film grown by thermal oxidation on different silicon substrates. A slightly dispersive Michelson interferometer with one of its mirrors replaced by a thin-film structure is used to measure the reflectance and interferometric phase of the thin-film structure at the same time. The experimental data are used to determine precisely thickness of the SiO_2 thin film on silicon wafers of two crystallographic orientations and different dopant concentrations. We confirmed very good agreement between the experimental data and theory and revealed that the thin-film thickness, which varies with the type of silicon substrate, depends linearly on the wavelength at which minimum in the spectral reflectance occurs. Similar behaviour was revealed for the interferometric phase.

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1 Introduction

The physical thicknesses and wavelength dependences of optical constants are the fundamental parameters and characteristics of thin-film structures. There are many optical methods, based on ellipsometric [1,2], reflectometric [3], or interferometric [4–6] measurements usable to determine the parameters and characteristics. Ellipsometric measurements performed at a single wavelength and a fixed angle of incidence provide the film thickness and optical constants [1]. Measurements by spectroscopic ellipsometry provide the results over a wide wavelength range with greater precision and accuracy [2]. Normal incidence spectroscopic reflectometry [3] applied over a wide wavelength range is a useful tool for the characterization of thin films and multilayer structures commonly encountered in semiconductor industry.

The optical method most commonly employed for micrometer-scale thickness measurements is Fourier transform infrared [4] and white-light [5] interferometry. The use of white-light interferometry was extended into the spectral domain [6,7] where the phase of the reflected wave, which changes as a function of wavelength [8] and layer thickness, is inscribed in the recorded spectral interferogram.

Recently, we used dispersive white-light spectral interferometry for measuring the thickness of SiO_2 thin film on a silicon wafer [9]. The technique utilizes a slightly dispersive Michelson interferometer with one of the mirrors replaced by a thin-film structure of known optical constants. The thickness of the thin film is determined from the fit of the recorded spectral interferogram to the theoretical one. More recently, the use of dispersive white-light spectral interferometry was extended for measuring the thin-film thickness utilizing the absolute phase retrieval from the spectral interferogram [10]. However, the results suffered from the systematic phase errors due to the optical components present in the interferometer. To minimize them, we applied a procedure with the reference measurement [11].

In this paper, we extend the use of spectral reflectometry and interferometry for measuring small changes in thickness of SiO_2 thin film grown by thermal oxidation on different silicon substrates. The reflectance and interferometric phase (nonlinear-like phase [10,11]) of a thin-film structure, which are measured in a slightly dispersive Michelson interferometer at the same time and which agree very well with theory, are used to determine precisely thickness of the SiO_2 thin film on silicon wafers of two crystallographic orientations and different dopant concentrations. We revealed that the thin-film thickness, which varies with the type of silicon substrate, depends linearly on the wavelength at which minimum in the spectral reflectance occurs. Similarly, we revealed the

Correspondence to: P. Hlubina, Phone: 420 597 323 134, Fax: 420 597 323 139, E-mail: petr.hlubina@vsb.cz

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thin-film thickness depends linearly on the wavelength at which minimum in the interferometric phase occurs.

2 Experimental methods

We used different procedures for measurement of both the reflectance and nonlinear-like phase function of a thin-film structure in the same experimental set-up employing a dispersive Michelson interferometer and a spectrometer [11].

2.1 Method of spectral reflectometry

The procedure for the reflectance measurement in a dispersive Michelson interferometer is with the blocked one of its arms and it consists of three steps: first by blocking the source, the background spectrum $I_{bkg}(\lambda)$ is measured, second by using a reference sample instead of one of the mirrors of the interferometer, the reference reflection spectrum $I_{ref}(\lambda)$ is measured, and third by using the thin-film structure instead of the reference sample, the reflection spectrum $I_{meas}(\lambda)$ of the thin-film structure is measured. The absolute reflectance $R(\lambda)$ of the thin-film structure is given by

$$R(\lambda) = \frac{I_{meas}(\lambda) - I_{bkg}(\lambda)}{I_{ref}(\lambda) - I_{bkg}(\lambda)} R_{ref}(\lambda), \qquad (1)$$

where $R_{ref}(\lambda)$ is the theoretical reflectance of the reference sample.

2.2 Method of spectral interferometry

The procedure for the interferometric measurement [10, 11] is based on recording the spectral interferograms in a dispersive Michelson interferometer with one of its mirrors replaced by a thin-film structure and retrieving the absolute optical path difference (OPD) $\Delta(\lambda)$. The OPD $\Delta(\lambda)$ is used to construct for a chosen mirror position $L = L_0$ the nonlinear-like phase function $\delta(\lambda)$ given by the relation

$$\delta(\lambda) = (2\pi/\lambda)[2L_0 + 2n(\lambda)t_{\text{eff}} - \Delta(\lambda)] + \delta_2(\lambda), \qquad (2)$$

where t_{eff} is the effective thickness of a beam splitter cube [9–11] and $\delta_2(\lambda)$ is the phase change on reflection from the mirror in the interferometer. To compensate for the phase change $\delta_2(\lambda)$, a next measurement step with the reference sample used instead of the thin-film structure needs to be applied [11].

3 Experimental set-up

The experimental set-up used in the application of spectral reflectometry and interferometry to analyse the effect of silicon substrate on the thickness of SiO_2 thin film is the same as that shown in previous papers [9–11]. It consists of a white-light source: a halogen lamp HL-2000 (Ocean Optics) with launching optics, an optical fibre with a collimating lens, a bulk-optic Michelson interferometer with a cube beam splitter made of BK7 optical glass, a metallic mirror connected to a micropositioner, a thin-film structure, a microscope objective, micropositioners, a read optical fibre, a miniature fibre-optic spectrometer S2000 (Ocean optics), an A/D converter and a personal computer.

The thin-film structure is represented by a uniform SiO_2 thin film on a silicon wafer. Seven samples of different silicon substrates affecting SiO_2 thin-film thickness were under study. The SiO_2 thin films on the silicon wafers were prepared using a dry oxidation process described by the so-called Deal-Grove model [12]. Single-crystal silicon wafers from ON Semiconductor, Czech Republic, were characterized by subsequent parameters: diameter (100±0.5) mm, B doped type P of different dopant concentrations (samples 1 to 3) with wafer orientation (111) and P doped type N of different dopant concentrations (samples 4 to 7) with wafer orientation (100) and thickness (381±25) μm. Before the oxidation, the wafers were cut into 40×40 mm squares, cleaned by standard methods and then annealed in a furnace at 1200°C. According to the model, the annealing time was selected in order to prepare SiO_2 thin film with thickness of approximately 300 nm.

4 Experimental results and discussion

First, samples with both (111) and (100) oriented silicon wafers and the lowest or the highest dopant concentration were measured. For these samples, the optical parameters of the silicon substrate (the refractive index and the extinction coefficient) as a function of wavelength were known from the ellipsometric measurements. The reflectance of sample 1 with the SiO_2 thin film on the silicon wafer of orientation (111) and the lowest dopant concentration was measured by the three-step procedure presented above. Figure 1 shows a comparison of the results obtained by fitting the theoretical reflectance $R(\lambda)$ to the measured one $R^e(\lambda)$ using the Levenberg-Marquardt least-squares algorithm. The method determines the maximum-likelihood estimate of the thin-film thickness d that minimizes the figure-of-merit function χ^2, defined by

$$\chi^2(d) = \sum_{i=1}^{N}[R^e(\lambda_i) - R(\lambda_i; d)]^2, \qquad (3)$$

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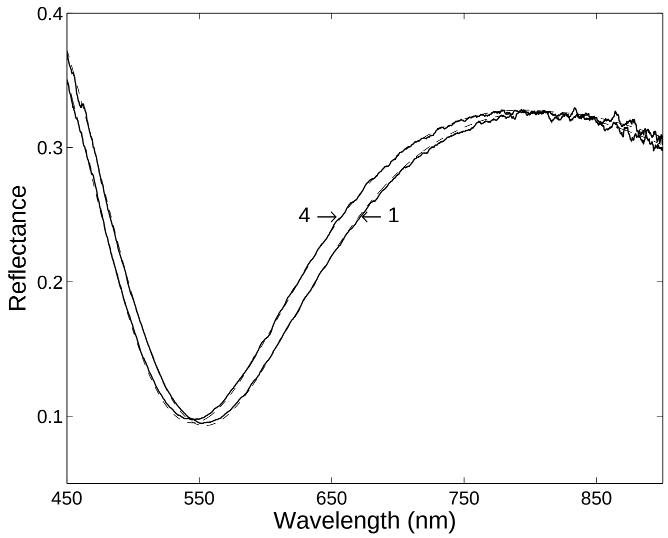

Fig. 1 The measured reflectance as a function of wavelength with the corresponding fit (dashed) for samples 1 and 4

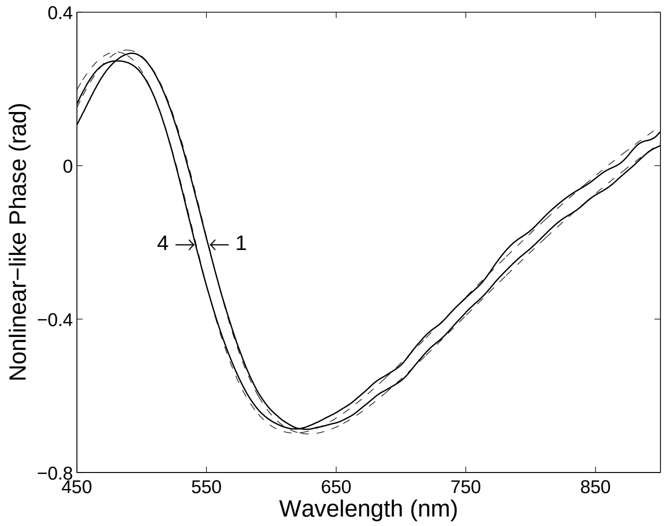

Fig. 2 The measured nonlinear-like phase as a function of wavelength with the corresponding fit (dashed) for samples 1 and 4

where λ_i are wavelengths at which the fit was performed (450 to 900 nm). Theoretical values of the reflectance $R(\lambda)$ were computed according to the well-known relations that take into account the known wavelength dependence for the refractive index of SiO_2 thin film [9,10] and the measured complex refractive index of the silicon substrate. Figure 1 demonstrates very good agreement between theory and experiment with the correlation coefficient as high as 0.99965 and the thin-film thickness d=280.1 nm.

The thickness d of the SiO_2 thin film on the silicon substrate was also measured by the technique of spectral interferometry. The OPD $\Delta(\lambda)$ retrieved from the recorded spectral interferograms [13] for sample 1 and the known phase function $\delta_2(\lambda)$ [11] were used in Eq. (2) for precise determination of the nonlinear-like phase $\delta(\lambda)$. The function was compared with theory in order to determine the thin-film thickness d. Figure 2 shows a comparison of the results of fitting the theoretical nonlinear-like phase $\delta(\lambda)$ to the measured one $\delta^e(\lambda)$ using the Levenberg-Marquardt least-squares algorithm. The method determines the maximum-likelihood estimate of the thin-film thickness d that minimizes the figure-of-merit function χ^2, defined in a similar way as that in Eq. (3). Figure 2 demonstrates very good agreement between theory and experiment with the correlation coefficient as high as 0.99914 and the thin-film thickness d=280.1 nm. The thicknesses of the SiO_2 thin film obtained by both measurement techniques are the same.

The measured reflectance of sample 4 with the SiO_2 thin film on the silicon wafer of orientation (100) and the lowest dopant concentration is compared in Fig. 1 with the theoretical reflectance obtained by using the fitting algorithm presented above. Figure 1 demonstrates very good agreement between theory and experiment with the correlation coefficient as high as 0.99962 and the thin-film thickness d=275.1 nm. Moreover, Fig. 1 shows that the minimum in reflectance for sample 4 is shifted to shorter wavelengths in comparison with that for sample 1. Similarly, the measured nonlinear-like phase function of the same sample is compared in Fig. 2 with the theoretical phase function obtained by using the fitting algorithm presented above. Figure 2 demonstrates very good agreement between theory and experiment with the correlation coefficient as high as 0.99917 and the thin-film thickness d=275.2 nm. Moreover, similarly as in Fig. 1, the minimum in phase function for sample 4 is shifted to shorter wavelengths in comparison with that for sample 1.

We also measured the reflectance and the nonlinear-like phase function for sample 3 with the SiO_2 thin film on the silicon wafer of orientation (111) and the highest dopant concentration. These functions were compared with theoretical ones in order to determine the thin-film thickness d. The results obtained are shown in Table 1 demonstrating via the correlation coefficients very good agreement between theory and experiment. Similarly, Table 1 shows the thin-film thicknesses obtained from a comparison of the theoretical reflectance and nonlinear-like phase function with the measured ones for sample 7 with the silicon wafer of orientation (100) and the highest dopant concentration.

We can conclude from the obtained results that there is very good agreement between experiment and theory. The agreement refers to both the reflectance and nonlinear-like phase function. The correlation coefficients R_R and R_{NLP} indicate minimum discrepancy between the real structure and the theoretical model adopted. We also studied the effect of the model with the standard silicon substrate of the known complex refractive index

Table 1 The thicknesses d_R and d_{NLP} of the SiO_2 thin films with the corresponding correlation coefficients R_R and R_{NLP} for different types of silicon substrate with dopant concentration N_D

Sample No.	Type	N_D (cm^{-3})	d_R (nm)	R_R	d_{NLP} (nm)	R_{NLP}
1	P (111)	1.74×10^{15}	280.1	0.99965	280.1	0.99914
3	P (111)	1.31×10^{19}	278.6	0.99957	278.5	0.99908
4	N (100)	2.30×10^{15}	275.1	0.99962	275.2	0.99917
7	N (100)	3.16×10^{19}	276.6	0.99968	276.6	0.99906

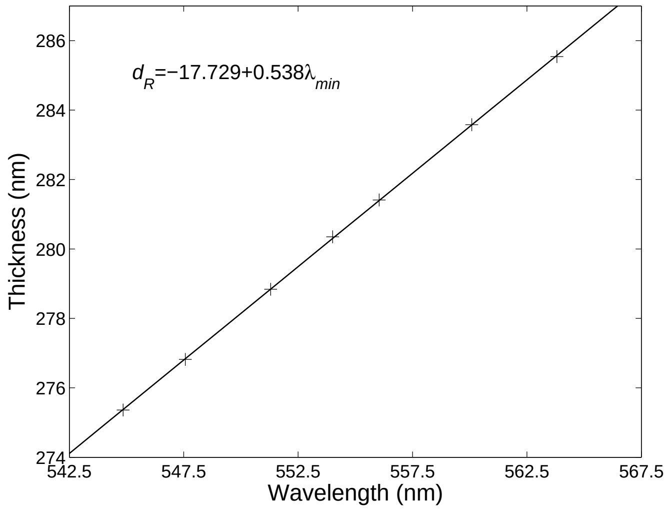

Fig. 3 Thickness d_R of the SiO_2 thin film on the silicon substrates as a function of the minimum wavelength λ_{min} in the reflectance

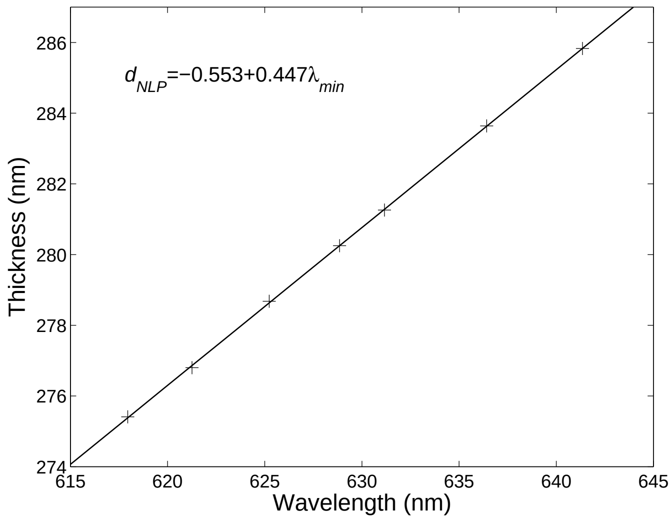

Fig. 4 Thickness d_{NLP} of the SiO_2 thin film on the silicon substrates as a function of the minimum wavelength λ_{min} in the nonlinear-like phase function

[14] on the thickness of the SiO_2 thin film. We revealed for all the four samples from Table 1 that the standard model gives all the thicknesses greater by a 0.2 nm in comparison with those for real substrates what represents systematic error below 0.1%. We can also conclude that the thickness of the SiO_2 thin film is greater for orientation (111) than for orientation (100) of silicon wafers of the comparable dopant concentration. This effect was also confirmed for sample 2 with the silicon wafer of orientation (111) and sample 6 with the silicon wafer of orientation (100), both with dopant concentrations about 8×10^{17} cm^{-3}. Using the model with the standard silicon substrate in processing the measured reflectance, sample 2 has a thickness of 285.5 nm and sample 6 has a thickness of 283.6 nm. Finally, the processing of the measured reflectance for sample 5 with the silicon substrate of orientation (100) and dopant concentration of 2.85×10^{16} cm^{-3} gives a thickness of 281.4 nm. Similar results were obtained by processing the measured nonlinear-like phase functions.

Figures 1 and 2 demonstrate the shift in the minimum of both the reflectance and nonlinear-like function with the thickness of the SiO_2 thin film. Both figures also confirm very good agreement between theory and experiment. Figure 3 shows the dependence of the thickness d_R of the the SiO_2 thin film on the position λ_{min} of the minimum in the theoretical reflectance evaluated by using the model with the standard silicon substrate of the known complex refractive index [14]. The dependence is linear and it enables us to determine the thin film thickness from the position of the minimum in the measured reflectance. If we assume that the precision in determining the minimum wavelength is 0.32 nm (wavelength distance of two adjacent pixels of spectrometer S2000), the error in measuring the thickness is 0.2 nm. Similarly, Fig. 4 shows the dependence of the thickness d_{NLP} of the the SiO_2 thin film on the position λ_{min} of the minimum in the theoretical non-linear like phase function evaluated by using the model with the standard silicon substrate. The dependence is linear and it enables us to determine the thin-film thickness from the position of the minimum in the measured non-linear like phase function with an error of 0.1 nm.

5 Conclusions

We extended the use of spectral reflectometry and interferometry for measuring small changes in thickness

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of SiO_2 thin film grown by thermal oxidation on different silicon substrates. We measured the reflectance and interferometric phase in a slightly dispersive Michelson interferometer at the same time and revealed that the thickness of SiO_2 thin film differs from substrate to substrate. For the substrates of the known complex refractive index, very good agreement with theory was confirmed and the thin-film thickness was determined precisely. The thickness of the SiO_2 thin film was also determined using the model of the standard silicon substrate and the systematic error not exceeding 0.1% was reached what means that the model is usable. Seven samples of silicon wafers with different dopant concentrations and two different crystallographic orientations were under study. We revealed that the thickness of the SiO_2 thin film is greater for (111) oriented than for (100) oriented silicon wafers of the comparable dopant concentration. This is in agreement with the well-known fact that the rate of growth of oxide film is faster with (111) oriented wafers than with (100) oriented ones. Moreover, we revealed that the thin-film thickness, which varies with the type of silicon substrate, depends linearly on the wavelength at which minimum in the spectral reflectance occurs. Similarly, we revealed the thin-film thickness depends linearly on the wavelength at which minimum in the interferometric phase occurs.

The results obtained serve as an illustration of the feasibility of simple techniques in measuring precisely small changes in thickness of the SiO_2 thin film on silicon substrate from the reflectance or the nonlinear-like phase function. The use of the methods can be extended for other structures, the optical parameters of which are known.

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